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IS 12107-3 (1987): Methods of chemical analysis of alumino-silicate refractory materials, Part 3: Determination of aluminium [MTD 13: Ores and Raw Materials]



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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINO SILICATE REFRACTORY MATERIALS

PART 3 DETERMINATION OF ALUMINIUM

1. Scope — This standard (Part 3) covers a method for determination of aluminium (as Al_2O_3) in alumino silicate refractory materials.

2. Determination of Aluminium by EDTA Method

2.1 Outline of the Method — Aluminium (also iron and titanium when present) is complexed quantitatively with EDTA by boiling with an excess of the reagent. The excess EDTA is back titrated with standard zinc acetate solution at pH 5.3 using xylenol orange indicator. The aluminium EDTA complex (also titanium EDTA complex, if present) on boiling with sodium fluoride liberates EDTA which when titrated with standard zinc acetate solution gives the amount of aluminium and titanium present in the solution. Due correction for titania is also carried out.

2.2 Reagents

2.2.1 EDTA solution (0.025 M) — Dissolve 9.31 g of EDTA (disodium salt) ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$) in 1 litre of water.

2.2.2 Methyl orange indicator solution — 0.05 percent (m/v) in ethanol.

2.2.3 Dilute ammonia solution — 1 : 2 (v/v).

2.2.4 Buffer solution (pH 5.3) — Dissolve 21.5 g of sodium acetate with 2 ml of glacial acetic acid in 300 ml of water and dilute to 1 litre.

2.2.5 Xylenol orange indicator solution 0.1 percent (m/v) — Dissolve 0.1 g of xylenol orange indicator in 100 ml of water containing 2-3 drops of dilute hydrochloric acid (1 : 1).

2.2.6 Standard zinc acetate solution (0.025 M) — Dissolve 5.50 g of zinc acetate [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$] in 1 000 ml of water containing 25 ml of buffer solution (pH 5.3). Standardize against standard aluminium solution following the method for determination of aluminium as given in 2.3.1.

2.2.6.1 Standard aluminium solution (0.025 M) — Take an aluminium foil and wash it with absolute alcohol and dry in a desiccator. Weigh 0.674 5 g of aluminium foil into a beaker containing 25 ml of water and 25 ml of concentrated hydrochloric acid. Heat the solution to dissolve the metal. Cool and dilute the solution to 1 000 ml in a volumetric flask (1 ml = 0.001 275 g of Al_2O_3).

2.2.7 Sodium fluoride — Solid.

2.2.8 Dilute hydrochloric acid — 1 : 3 (v/v).

2.3 Procedure

2.3.1 Transfer 25 ml of main solution [see 2.3.3 of IS : 12107 (Part 2)-1987 'Methods of chemical analysis of alumino silicate refractory materials: Part 2 Determination of silica'] to a 250-ml conical flask. Add 35 ml of EDTA solution and 2-3 drops of methyl orange indicator solution. Add dilute ammonia solution dropwise to just yellow colour. Add 15 ml of buffer solution and boil for 5 minutes. Cool to room temperature. Add 5-6 drops of xylenol orange indicator and titrate the solution with standard zinc acetate solution to just pink colour. Add 1 g of sodium fluoride to the solution followed by 2-3 drops of dilute hydrochloric acid. Boil the solution again for 5 minutes. Cool to room temperature. Add 5 ml of buffer solution and titrate the released EDTA with standard zinc acetate solution using 5-6 drops of xylenol orange indicator to pink end point. The second titre (V ml) corresponds to the amount of titanium and aluminium.

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2.3.2 Calculation

$$\text{Aluminium (as Al}_2\text{O}_3 \text{), percent by mass} = \frac{V \times E \times 500 \times 100}{25 \times M} - (0.6738 \times T)$$

where

V = ml of zinc acetate consumed,

E = equivalent Al_2O_3 in g/ml of zinc acetate solution,

M = mass in g of sample taken, and

T = percent of TiO_2 by mass. [The value obtained by the method given in IS : 12107 (Part 5)-1987 'Methods of chemical analysis of alumino silicate refractory materials: Part 5 Determination of titanium'].

EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (Al_2O_3) and silica (SiO_2) in varying portions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour.

It is used in kilns, laddles and furnaces that operate at higher temperature or under conditions for which fireclay refractories are not suitable.

This Indian Standard has been prepared in different parts to cover the chemical analysis of various constituents in alumino silicate refractory materials. The other parts of the standard are:

- Part 1 Determination of loss on ignition
- Part 2 Determination of silica
- Part 4 Determination of phosphorus
- Part 5 Determination of titanium
- Part 6 Determination of iron
- Part 7 Determination of manganese
- Part 8 Determination of calcium and magnesium
- Part 9 Determination of sodium and potassium